Surface Buckling and Other High-throughput Measurement Techniques

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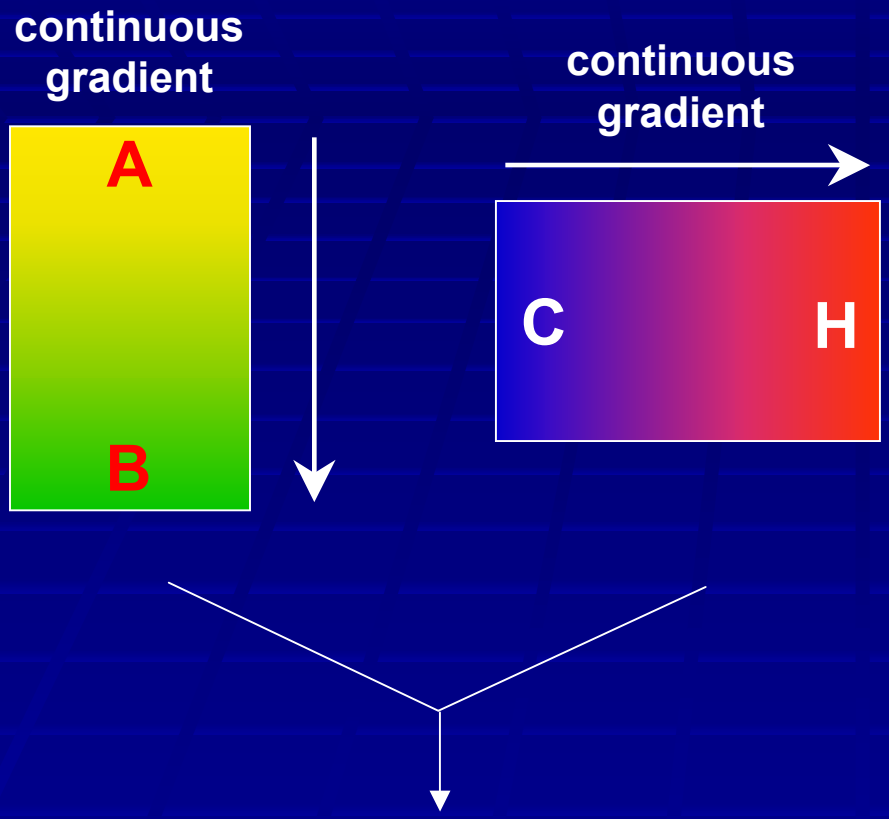
Combinatorial Material Science



gain access to every combination of composition and temperature

Gradient Toolkit

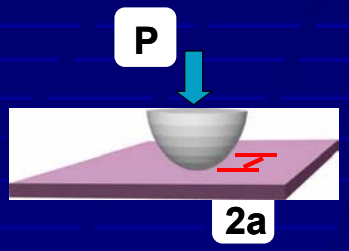
- Thickness
- Composition
- Temperature
- Surface energy
- UV exposure
- (Polymer Architecture)
- (Interfacial Tension)

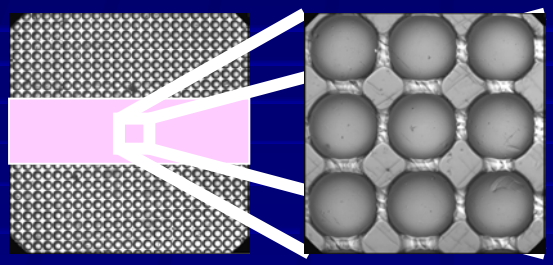
Areas of Interest

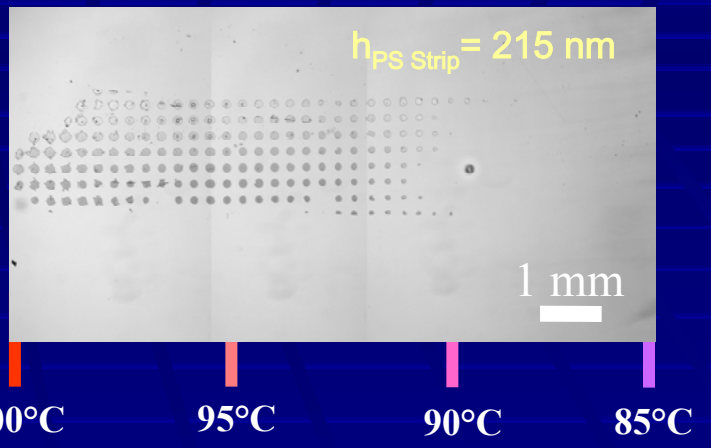
- Polymer dewetting
- Phase separation
- Copolymer ordering
- Polymer crystallization
- Cellular response to patterned surfaces

Combinatorial Methods at NIST

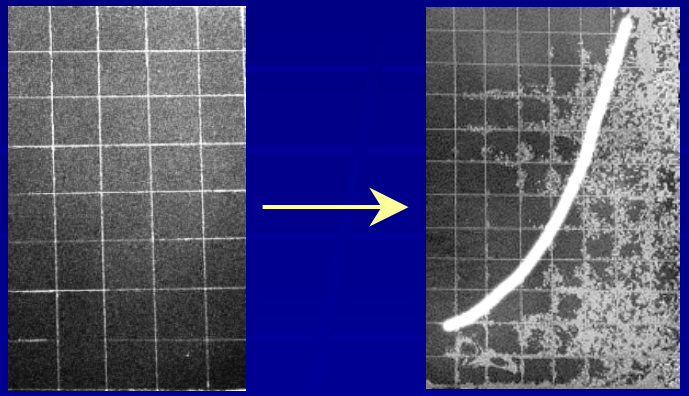
Multilens Contact Adhesion Test (MCAT)

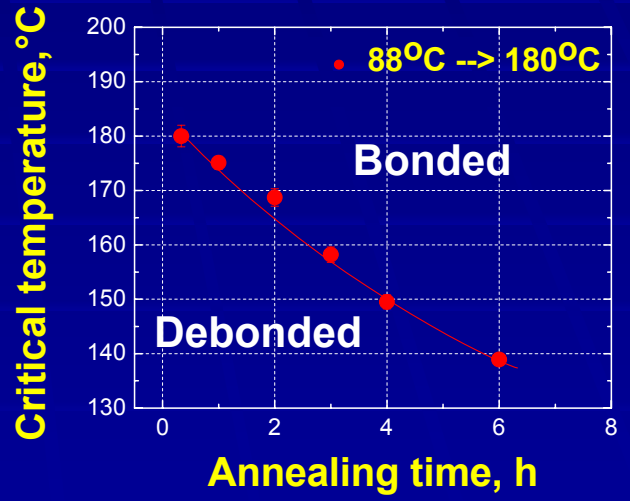






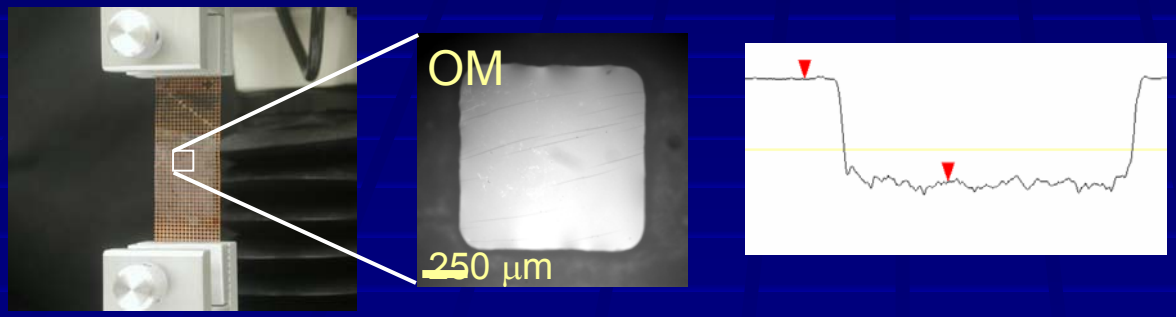
Combinatorial Edge-Debonding Test

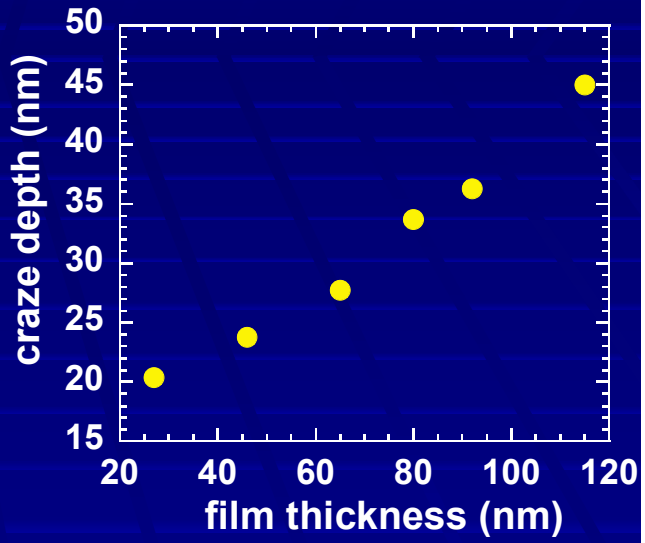




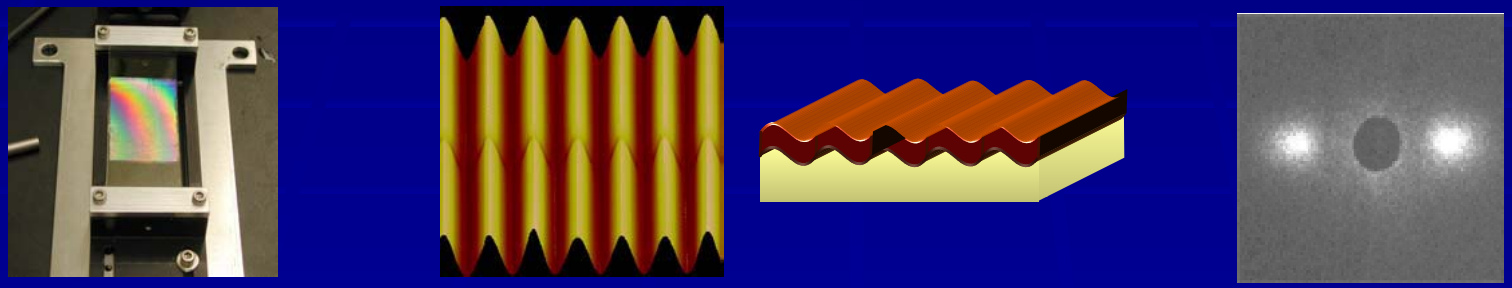
Combinatorial Methods at NIST

Copper Grid Test on Combinatorial Libraries





Buckling Instabilities for Mechanical Measurements



Outline

- *Thin films are increasingly being used in technological applications involving dielectric coatings, resist layers for lithography, electronic packaging, optical coatings, etc.*

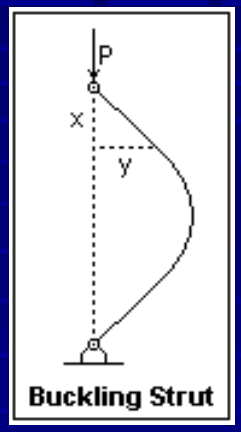
- *There is a growing need for test methods that can measure the mechanical properties (modulus, viscoelasticity, plastic deformation) of thin films since these material properties play a vital role in the ultimate performance of the end product.*

Background into Buckling Instabilities
The Making of a Measurement Platform
Sample Fabrication
Applications
Closing Thoughts / Directions

Surface Buckling - History

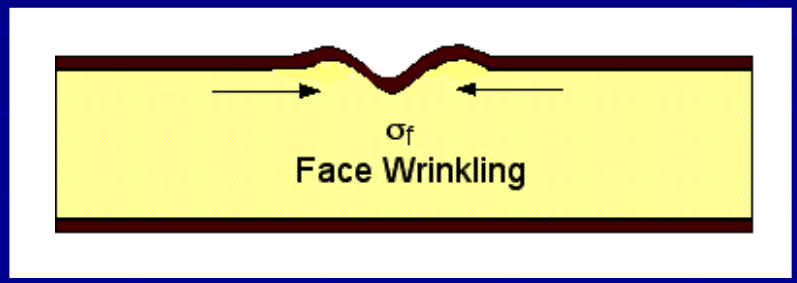
- **Euler : buckling of a beam or strut**

$$P_{CR} \approx \frac{EI}{L^2}$$



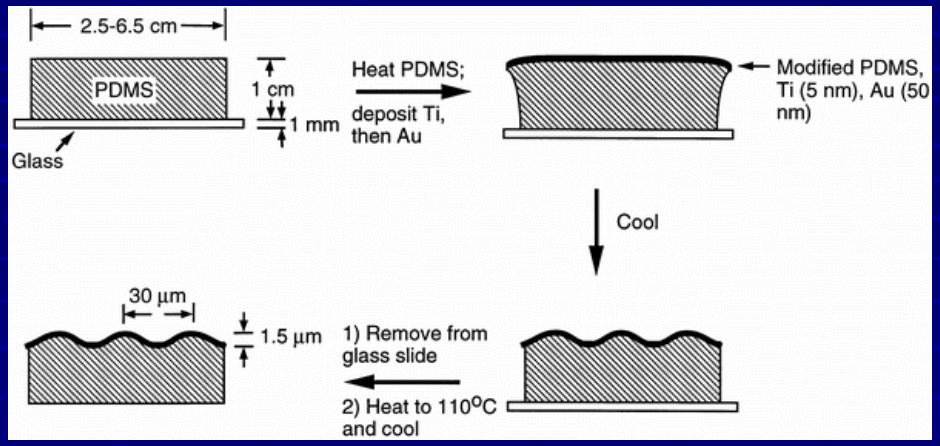
- **Five different modes of failure of a composite sandwich panel :**

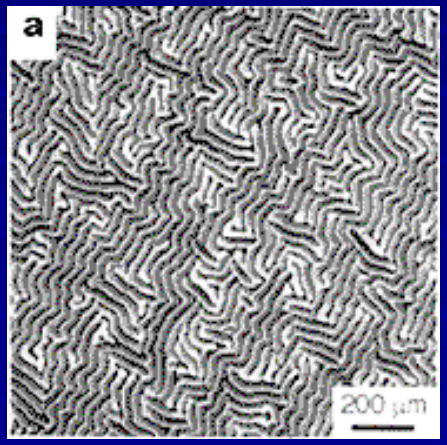
1. Yielding or fracture
2. **Buckling or wrinkling**
3. Core failure
4. Delamination
5. Indentation

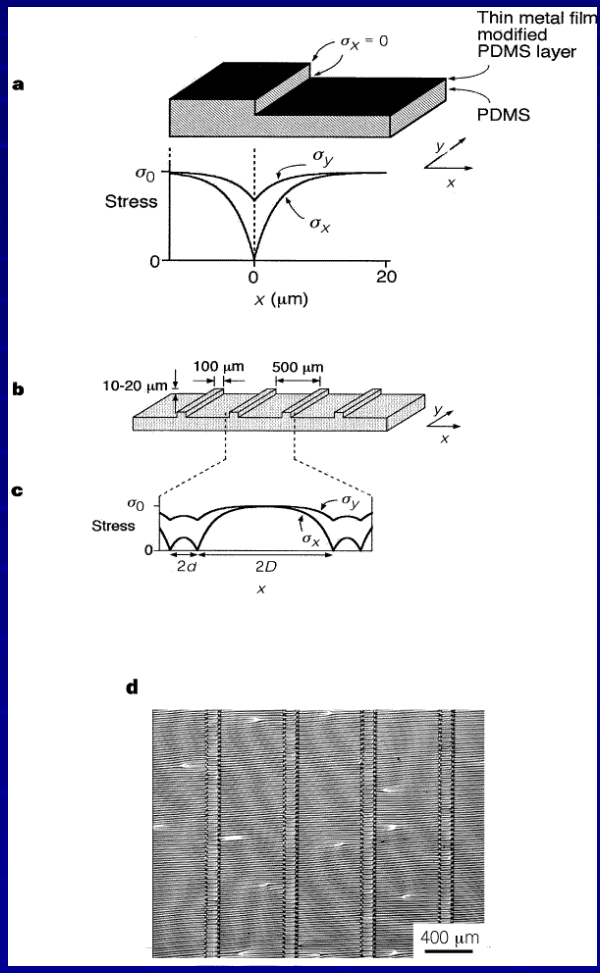


H.G.Allen, *Analysis and Design of Structural Sandwich Panels*, Pergamon Press, 1969.

Surface Buckling – Recent





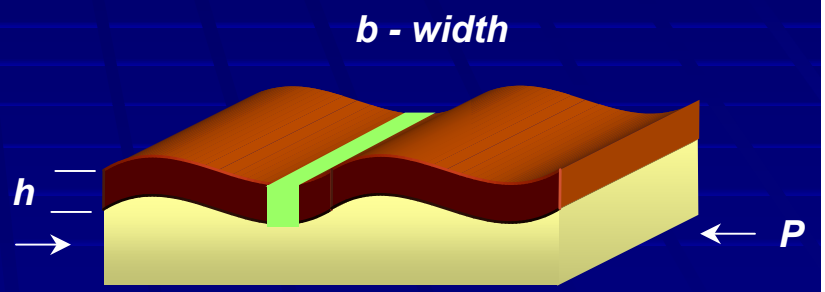


Bowden, Brittain, Evans, Hutchinson, & Whitesides. *Nature 393* (1998), 146.

Buckling of a Structural Sandwich

Let's start with a beam bending equation:

$$E_p b h^3 \frac{\partial^4 w}{\partial x^4} + P \frac{\partial^2 w}{\partial x^2} \approx \sigma_z(x) b$$



w(x) **– local displacement in the z-axis**
P **– compressive load/force**
b **– width of a section**
h **– thickness of the rigid plate**
$\sigma_z(x)$ **– shear stress**

Now, let's assume the sandwich buckles sinusoidally:

$$w = w_m \sin \frac{2\pi}{d} x$$

And, the necessary stress required is:

$$\sigma_z \approx -\frac{E_m w_m}{d} \sin \frac{2\pi}{d} x$$

H.G.Allen, *Analysis and Design of Structural Sandwich Panels*, Pergamon Press, 1969.

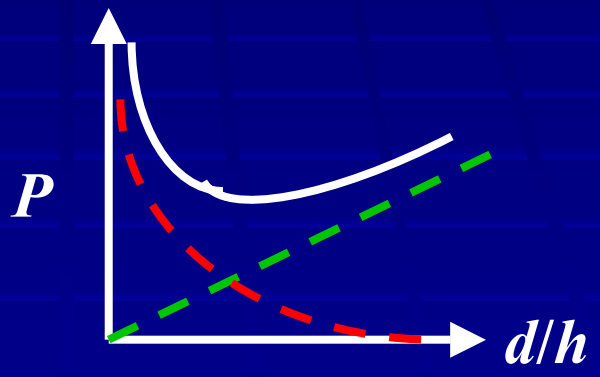
Buckling of a Structural Sandwich

Then, let's make all the necessary substitutions:

$$P \approx E_p \left(\frac{d}{h}\right)^{-2} + E_m \left(\frac{d}{h}\right)^{1}$$

What value of *d/h* will lead to a minimization of the lateral stress?

$$\frac{\partial P}{\partial (d/h)} = 0$$



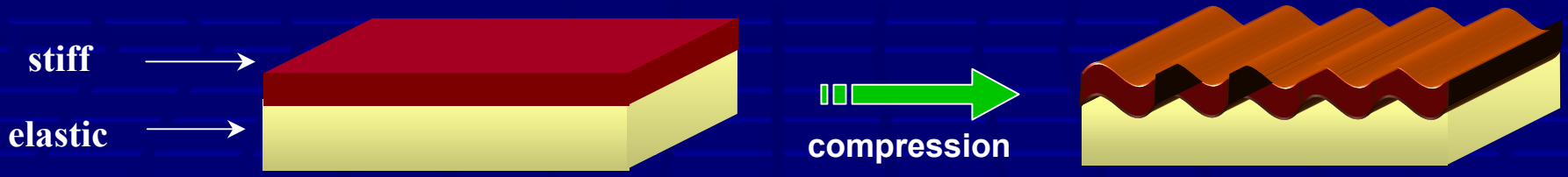
The answer is:

$$d \sim h\left(\frac{E_p}{E_m}\right)^{1/3}$$

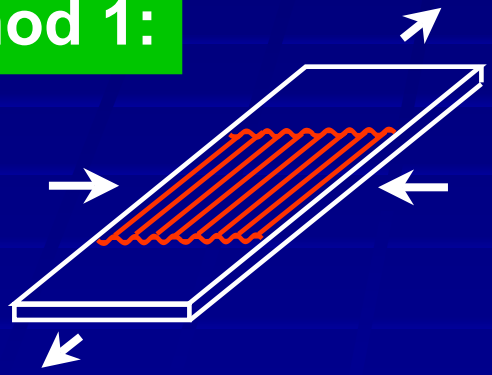
$$E_p = 12 \cdot E_m \cdot [(3-\upsilon)(1+\upsilon)]^{-1} (qh)^{-3}$$

H.G.Allen, *Analysis and Design of Structural Sandwich Panels*, Pergamon Press, 1969.

NIST Measurement Technique?



Method 1:



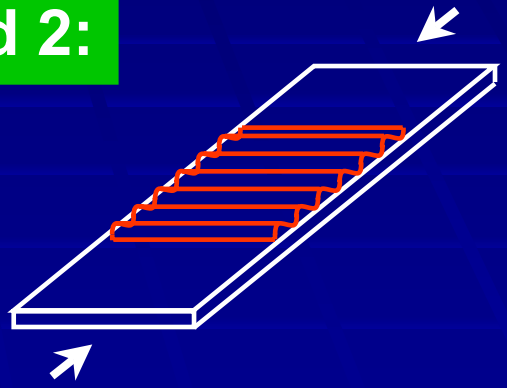
compression

$-\upsilon_m \varepsilon_y$

strain

ε_x

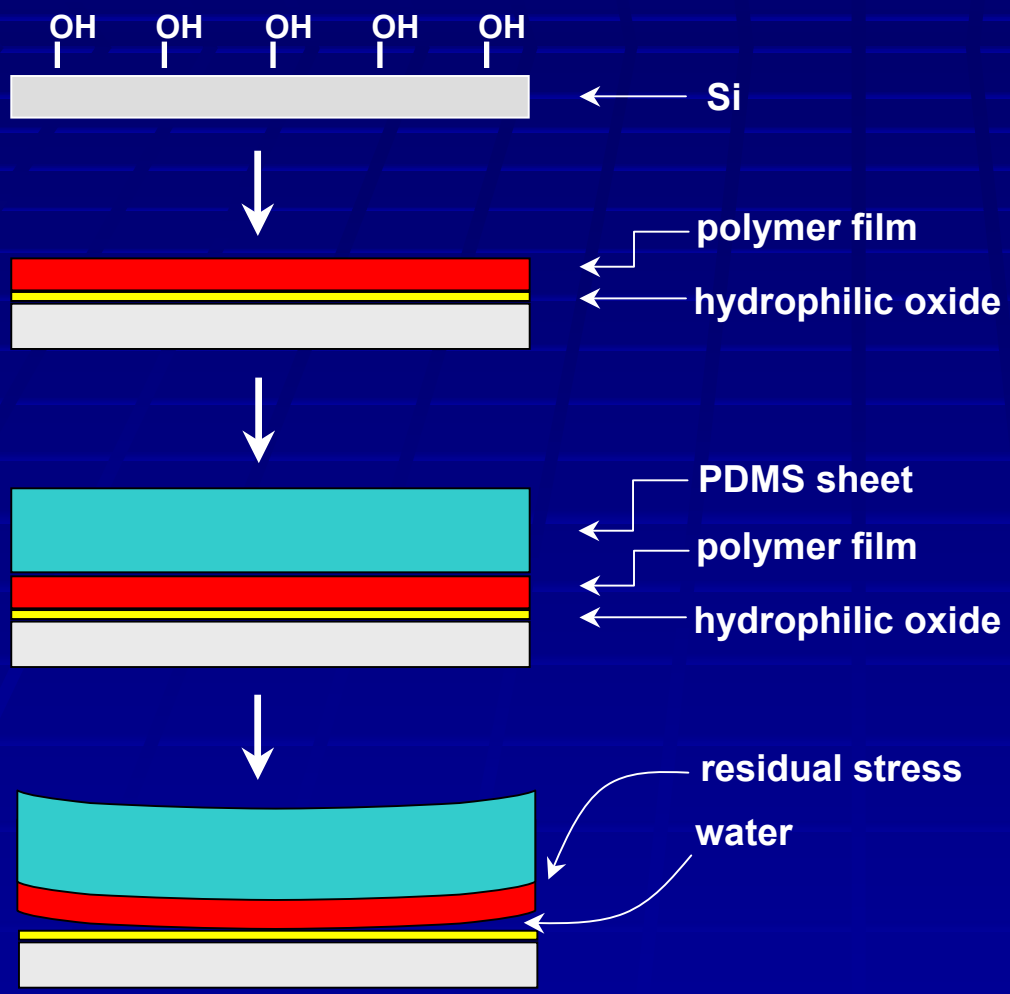
Method 2:



compression

ε_x

Sample Preparation



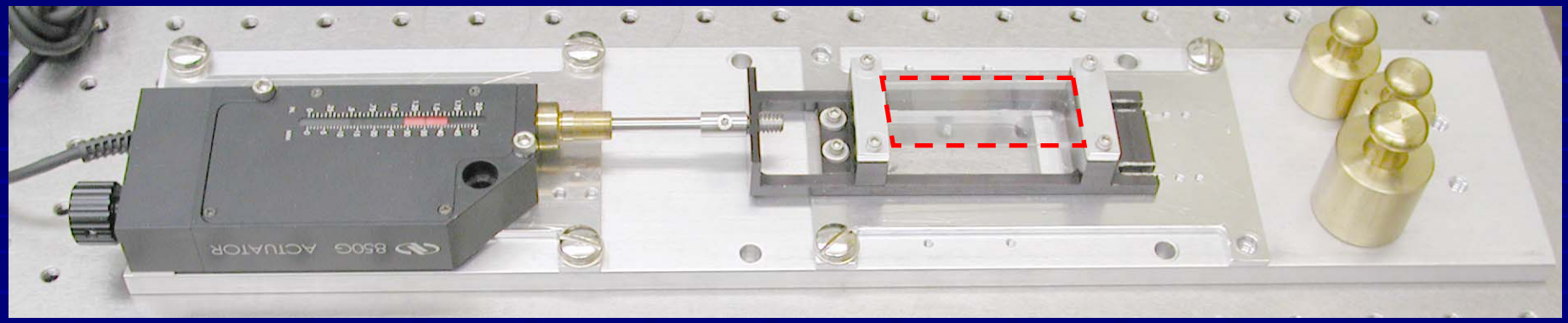
Make surface of silicon oxide hydrophilic

Prepare thin film by variety of techniques

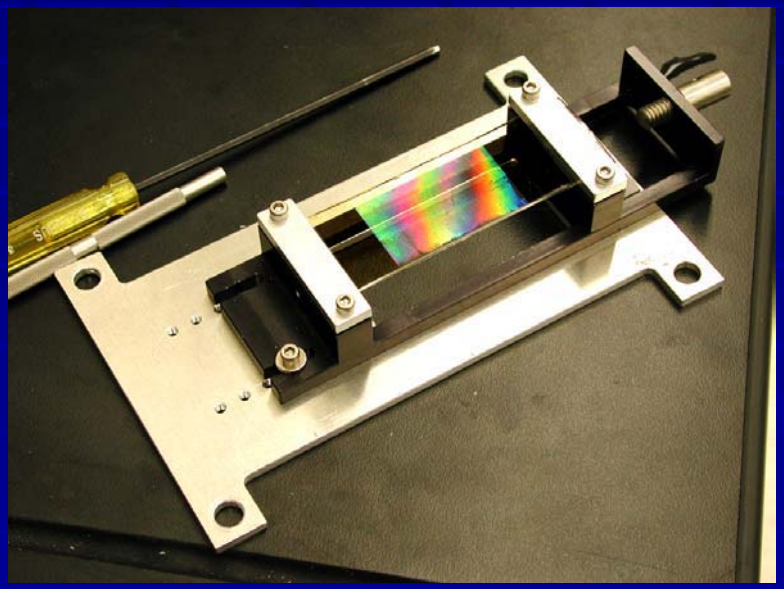
Cast PDMS sheet on top of substrate/film

Release laminate by immersing in water

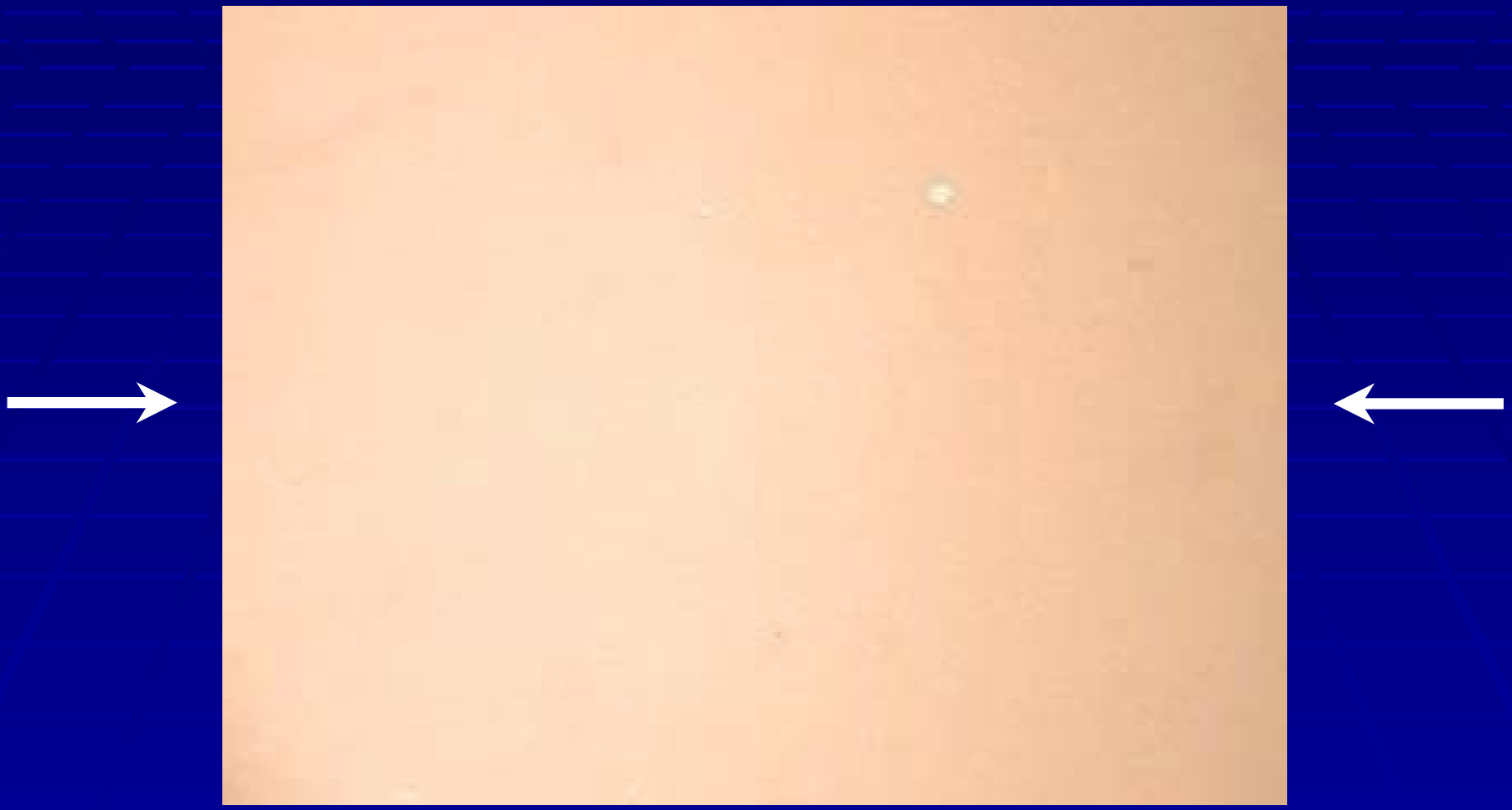
SIEBIMM – How?


ACTUATOR
850G


NIST
Polymers Division
100 Bureau Drive, Stop 8542
Gaithersburg, MD 20899-8542

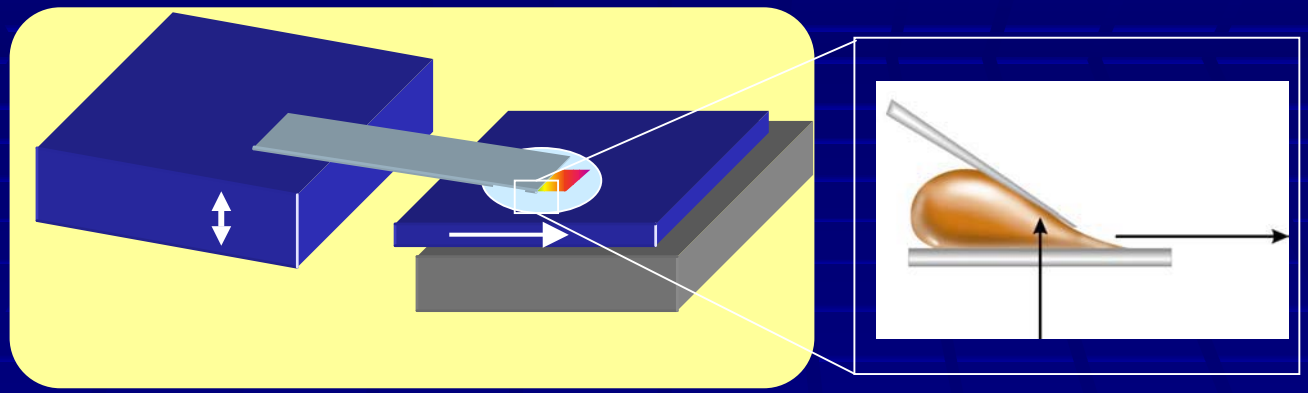


SIEBIMM - In Action



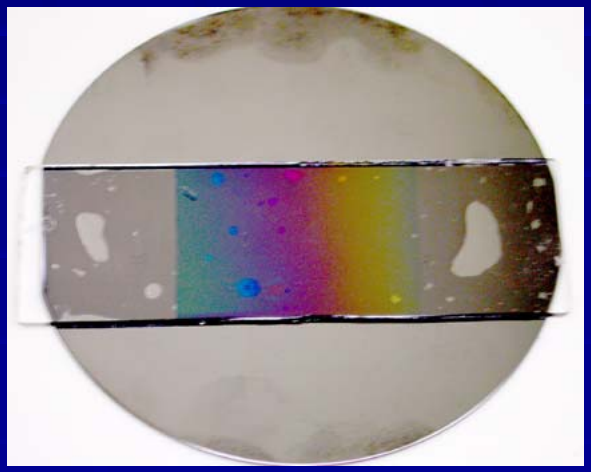
SIEBIMM - In Action

Gradient Thickness – Flow Coating

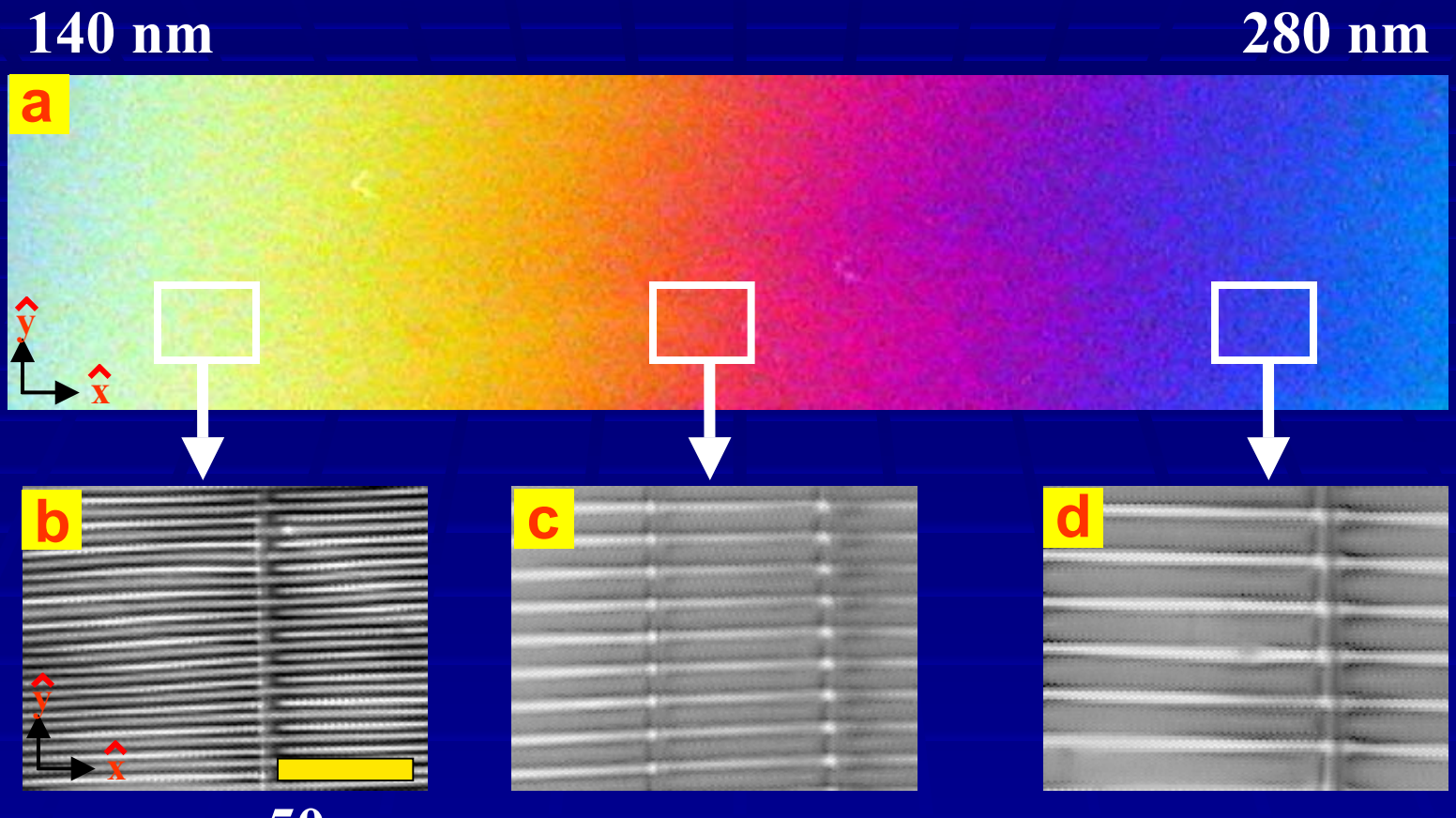


Why a thickness gradient?

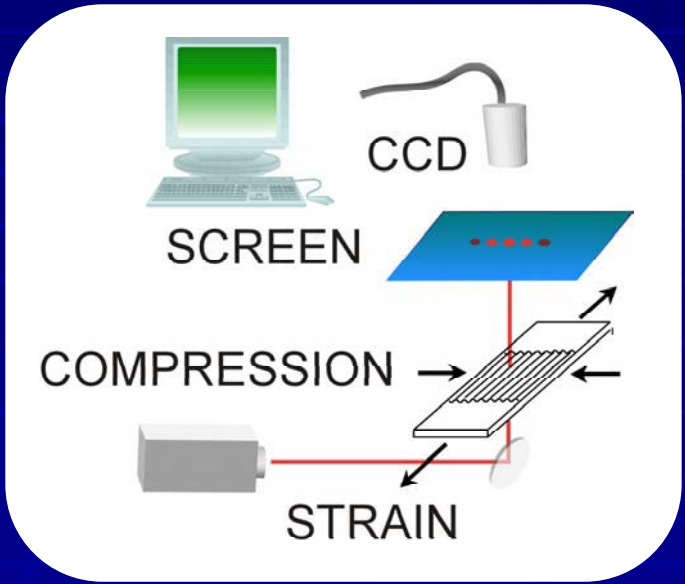
$$d \sim h\left(\frac{E_p}{E_m}\right)^{1/3}$$

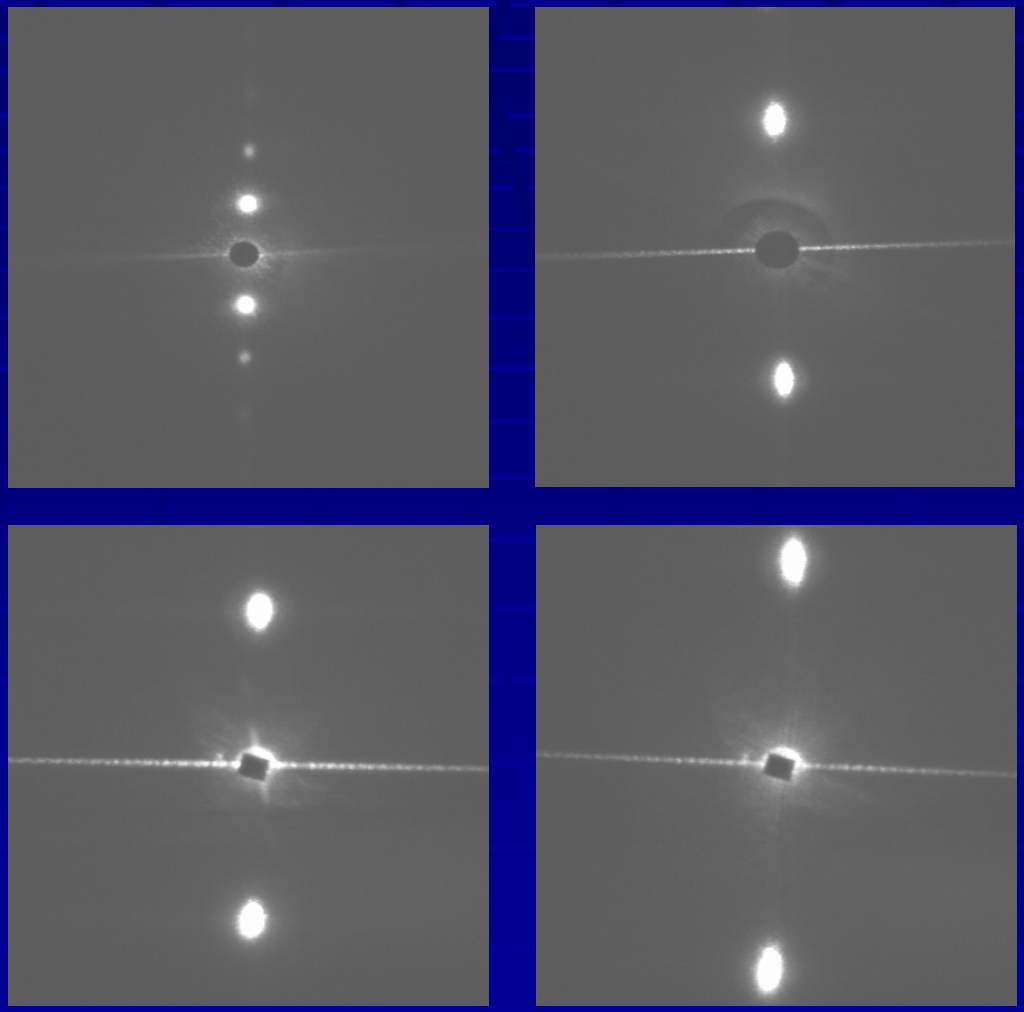


Gradient Thickness



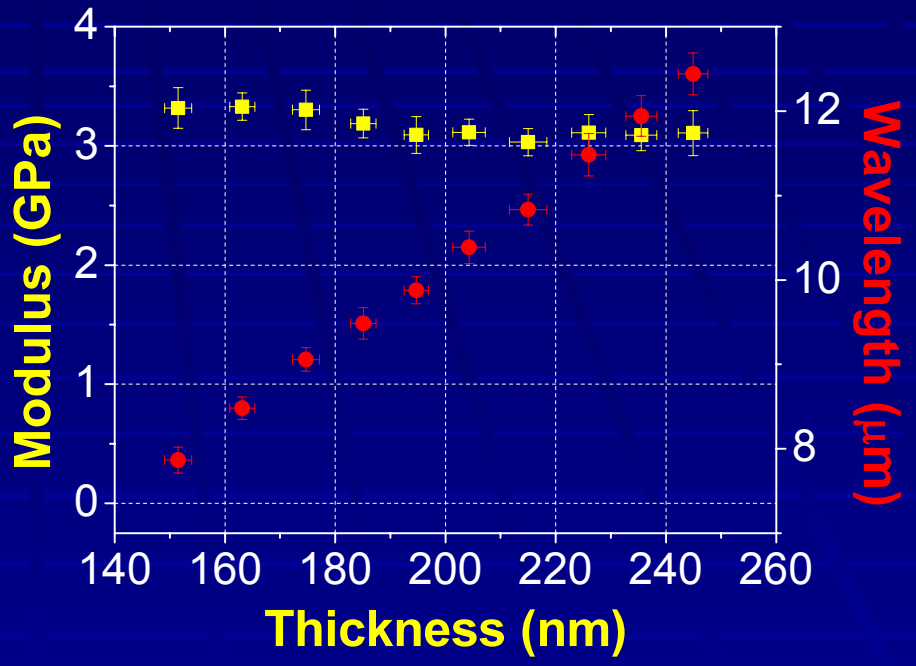
SALS Measurement





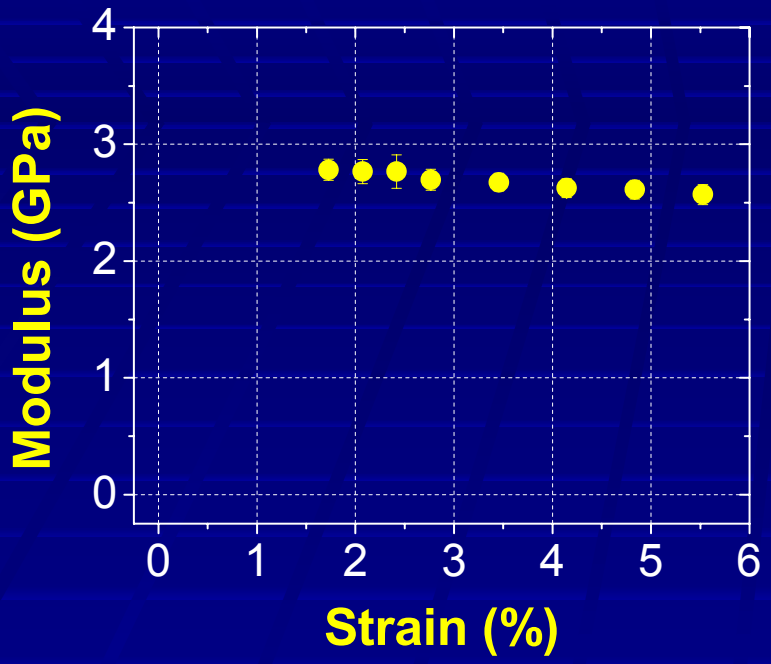
Gradient Thickness

- PS film with a thickness gradient.
- Buckling wavelength *d* increases linearly with thickness.
- SIEBIMM modulus:
  3.17 GPa ±0.11 GPa
- Flexural modulus:
  3.22 GPa ± 0.05 GPa
  (Donald Hunston, NIST)

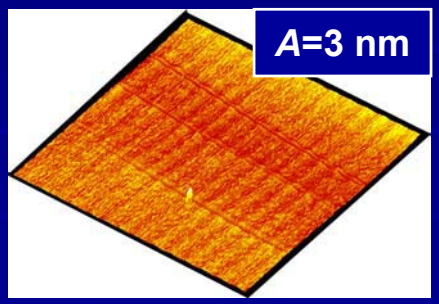


$$d \sim h\left(\frac{E_p}{E_m}\right)^{1/3}$$

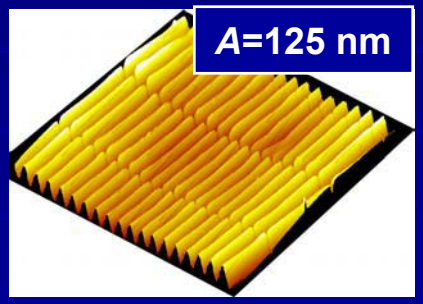
Effects of Strain



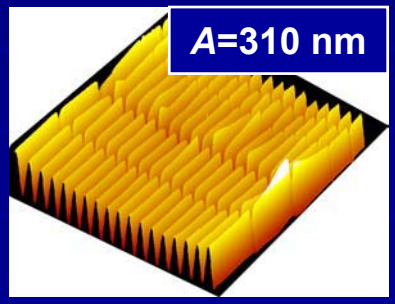
- Even for glassy/brittle polystyrene, modulus measurement relatively insensitive to strain (for strain < 5%).
- ε (critical) needed to induce buckling
- Measurements typically made at lowest strain that triggers instability.



3% strain



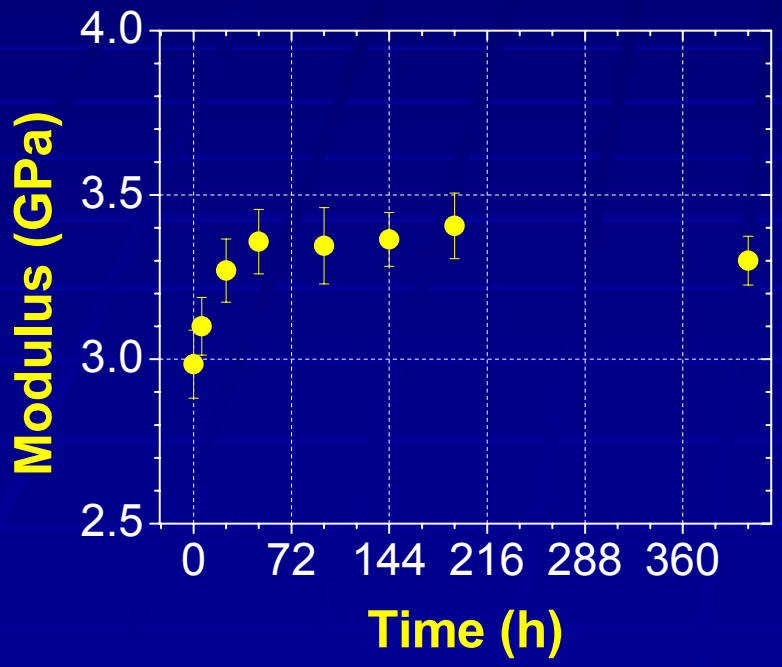
7% strain

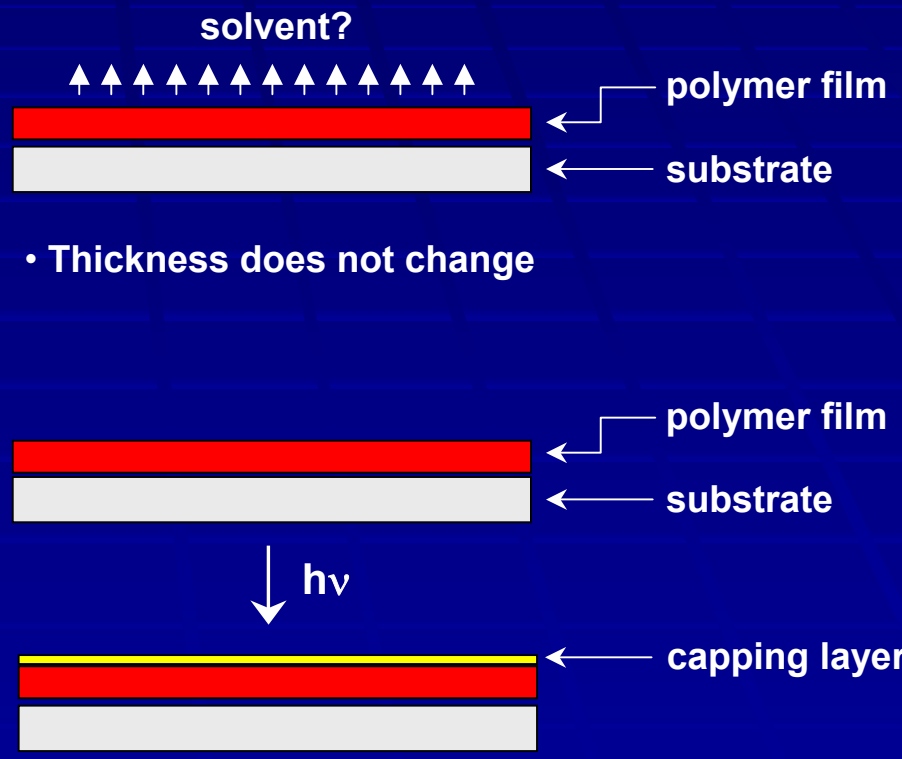


10% strain

Effects of Aging

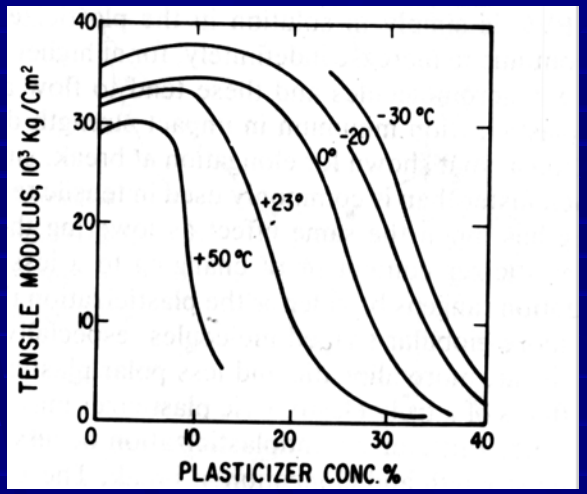
- Critical for defining protocols of a measurement technique.
- Properties of polymer film can change over time.
- Shows promise as a sensor.

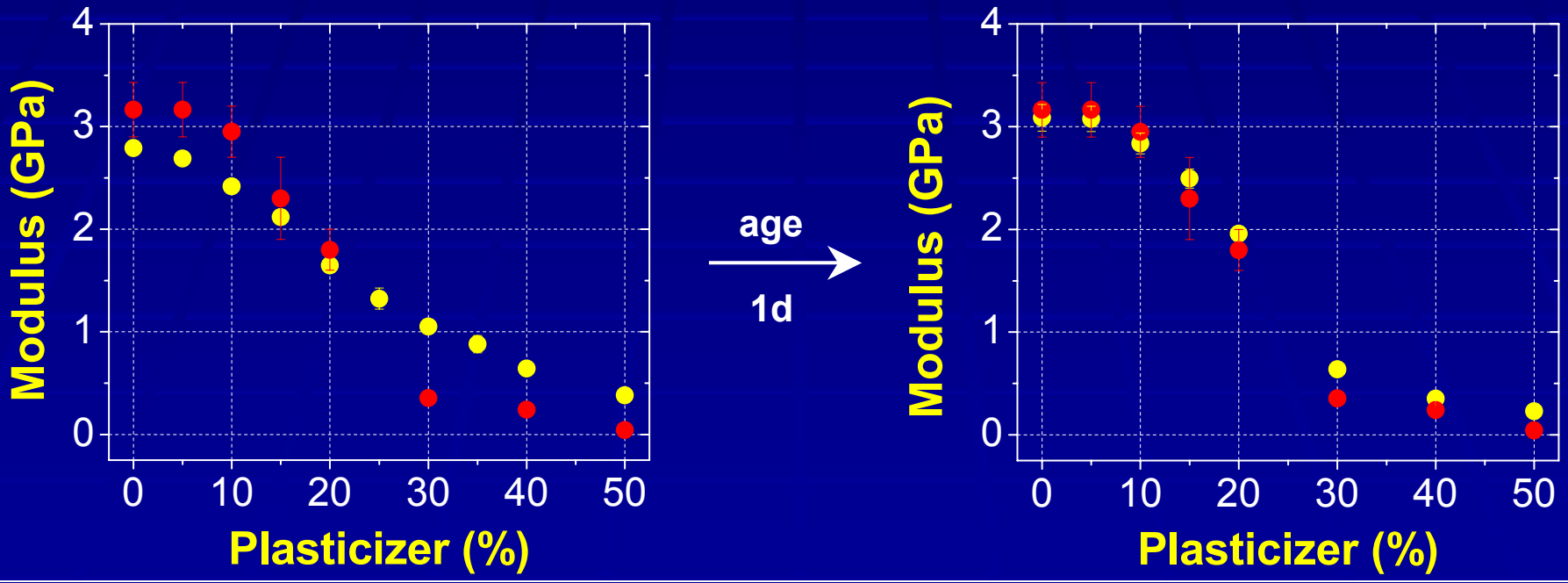




Plasticized Polymer Films

- Modulus measurement of plasticized polystyrene (dioctyl phthalate blend).
- SIEBIMM successfully follows decrease in modulus with plasticizer.
- Nanoindentation (red) agrees well with SIEBIMM. (Mark VanLandingham, NIST)





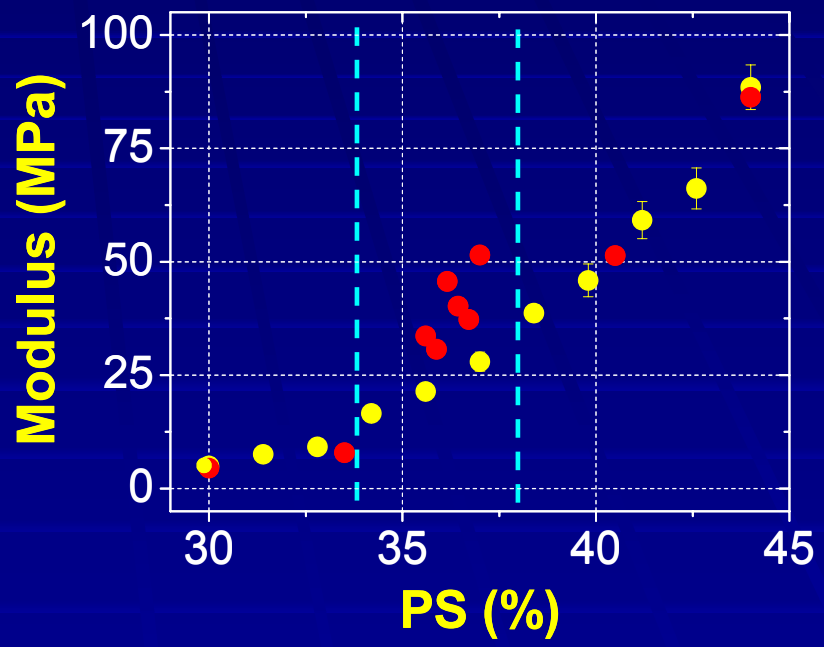
Softer Systems?

- Modulus of the film is tuned by ratio of two P(S-I-S) triblock copolymers:

Vector 4215 (30% PS, more rubbery)
Vector 4411 (44% PS, more glassy)

- Optically clear, miscible system

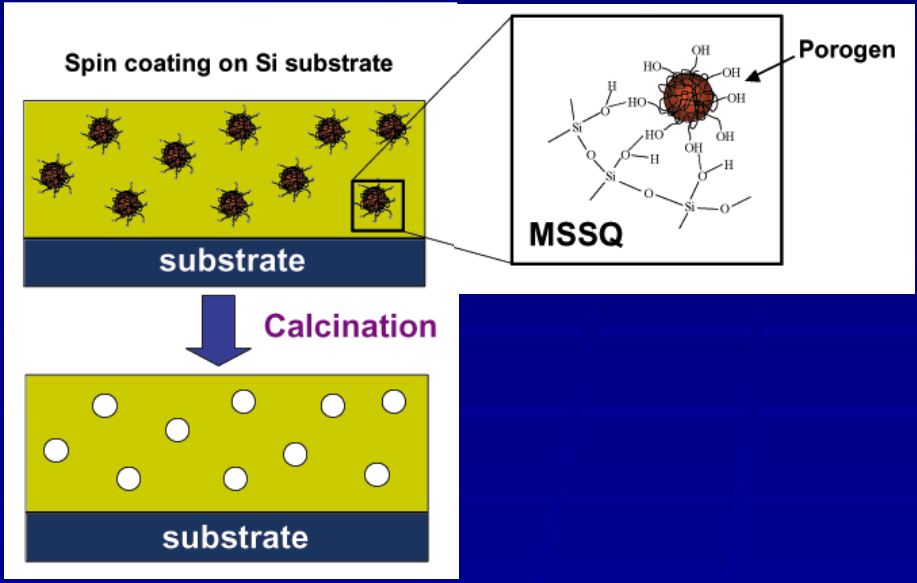
- We can span the range of polymeric E: GPa → MPa

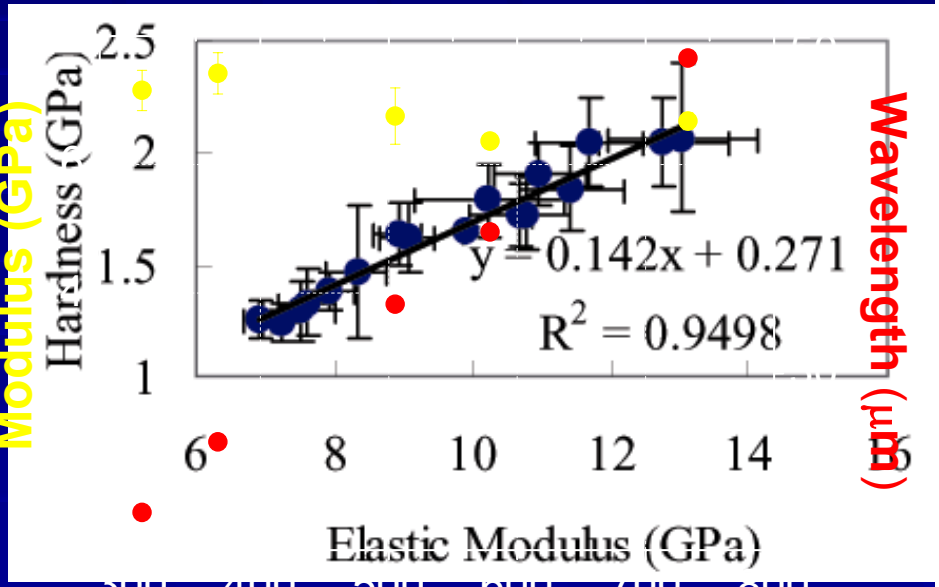


- K. Heffner, senior thesis, Princeton University. (Traditional Instron tests)

Nanoporous Films

- Nanoporous (low-k) films are a critical technology for sub-100 nm semiconductor applications.
- The mechanical properties (modulus, hardness, etc) are integral to the resilience of these films to CMP/planarization

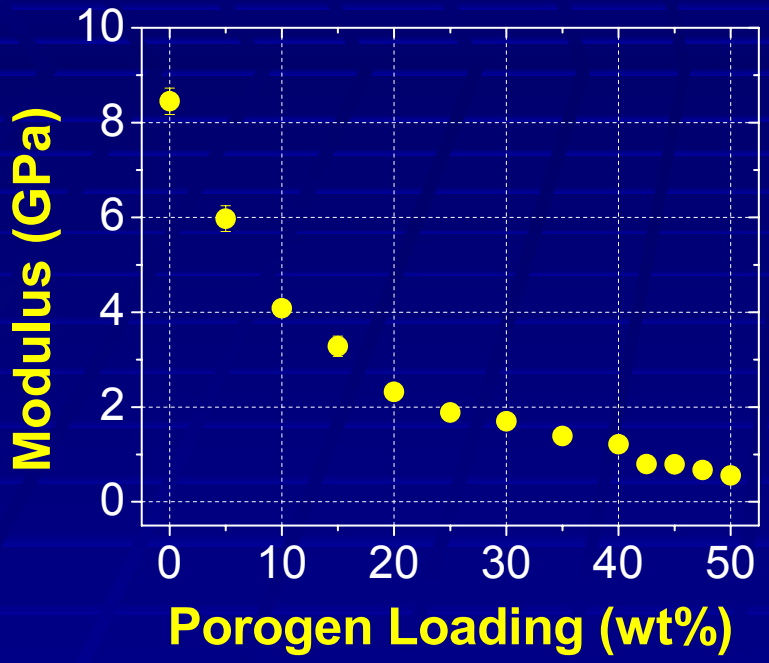


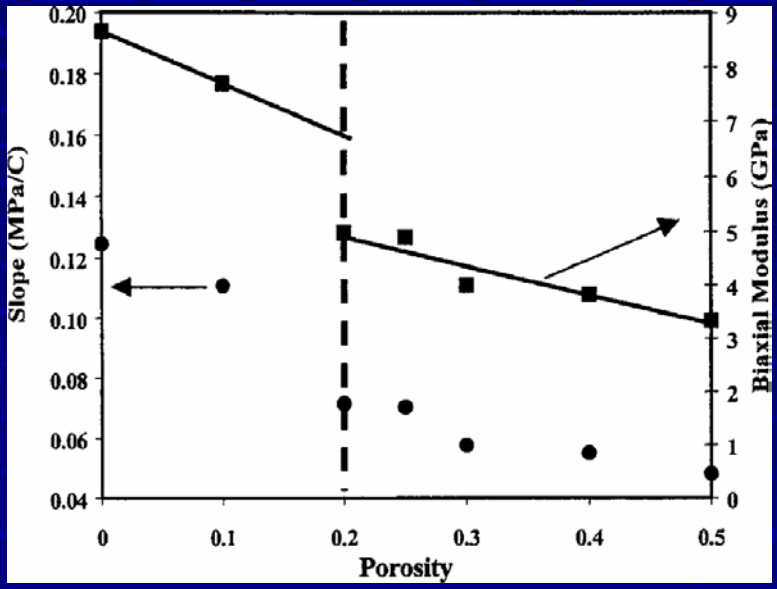


**Samples supplied by Dr. Kim, IBM Almaden*

Vella *et al., Mat. Res. Soc. Symp. Proc.* 716 B12.13

Nanoporous Films





Liu *et al., Appl. Phys. Lett.* 81 4180 (2002).

- Modulus decreases monotonically with increasing porosity.
- No abrupt transitions observed (percolation point).
- Highest loading leads to < 1 GPa modulus.

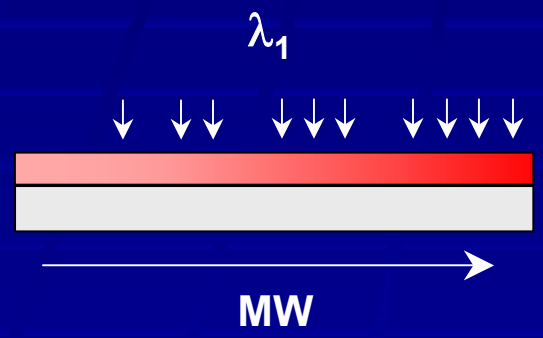
**Samples supplied by Dr. Kim, IBM Almaden*

Highlights and Thoughts

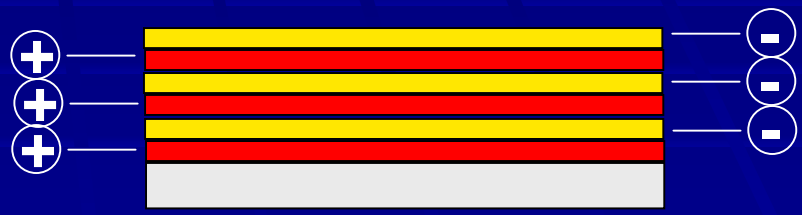
Mechanical Measurements

- screen materials
- technological applications
  - nanoporous
  - nanocomposites
- oscillatory strain
  - viscoelastic effects? (τ_R) → E' and E''
- step strain

- Tim Long – Reversible Macromolecules



- Paula Hammond – LBL Assemblies

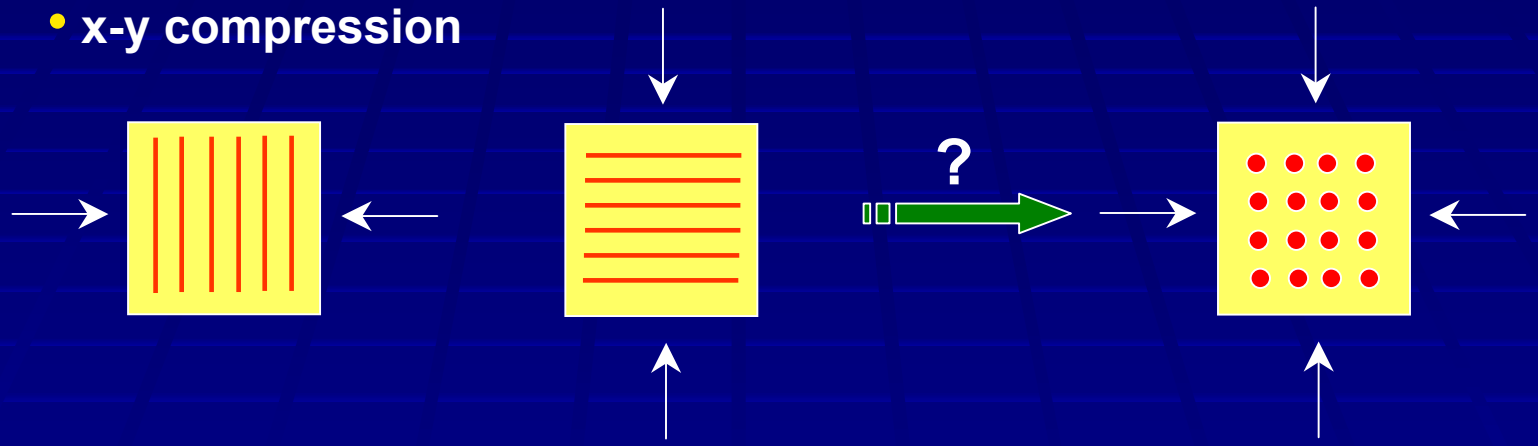


- Tom Russell – Long Range Order in BCPs

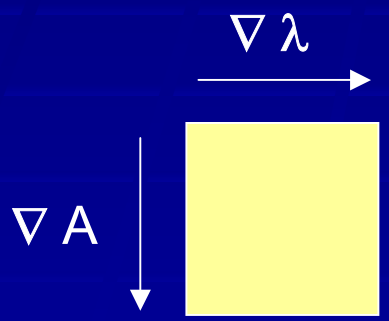
Highlights and Thoughts

Structural Manipulation

- x-y compression



- gradient thickness / strain



- oscillatory strain

  on-off switch for amplitude

Acknowledgments

- Alamgir Karim
- Eric Amis

- Jack Douglas
- Jan Groenewold
- Al Crosby

- Don Hunston
- Sheng Lin-Gibson
- Mark VanLandingham

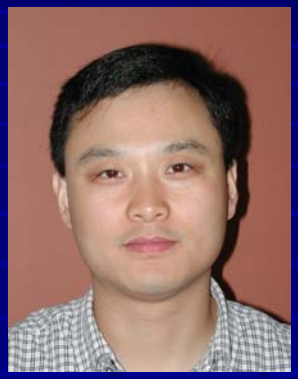
- National Research Council

- Craig Hawker / Kris Matyjaszewski
- GRC

Project Team

Team SIEBIMM:


Team CAD:




Team MCAT:



Management: